

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Applicants: Donald B. Appleby et al. : Group Art Unit: 1211

Serial No.: 08/360,184 : Examiner: E. White

Filed: December 20, 1994 :

For: **Polyol Polyester Synthesis**

DECLARATION UNDER 37 C.F.R. §1.608 OF JU-NAN KAO

Assistant Commissioner for Patents
Washington, DC 20231

Dear Sir:

I, JU-NAN KAO, declare that:

1. I am a co-inventor of and am familiar with the present Appleby et al U.S. patent application.

2. I received a Doctorate degree in Chemical Engineering from the City University of New York in 1987.

3. I am employed by the assignee of the present application, The Procter & Gamble Company, and have been working for The Procter & Gamble Company continuously since 1988. During my employment with The Procter & Gamble Company, I worked, inter alia, on developing a continuous process for the synthesis of polyol fatty acid esters, specifically sucrose

fatty acid esters, commonly referred to as sucrose polyesters, FG or FG base, by reaction of sucrose and fatty acid methyl esters.

4. From June 1988 to October 1989 I worked as a process engineer/researcher on the sucrose polyester reaction/process optimization project. My responsibilities included studying and optimizing process parameters, coordinating sucrose polyester laboratory experiments according to predetermined test plans, and analyzing the results, or having the results analyzed under my direction and control, according to established procedures.

5. I conducted and analyzed the results of a variety of sucrose polyester laboratory experiments between January 1989 and October 30, 1989. For each experiment which I conducted and which is described in this Declaration, I accurately recorded, on or about the day of the experiment, the general nature of the experiment, including pertinent reaction parameters, the results of the experiment, the date of the experiment and my signature in a Laboratory Notebook assigned to me for such a purpose.

6. During my employment with The Procter & Gamble Company, I have, from time-to-time, prepared and distributed biweekly and monthly reports accurately describing my activities, and/or activities of those under my direction and control, during the prior two week or monthly period which was the subject of the report. For each report, I accurately set forth therein the ending date for the prior two week or monthly period which was the subject of the report.

7. I have reviewed Exhibit 49 and confirm that it is an accurate copy of the Industrial Chemicals Product Development Biweekly Report which I prepared on or about February 8, 1989. As described in Exhibit 49, a minor side reaction which occurs during sucrose polyester synthesis is the formation of difatty ketone (DFK). DFK formation occurs particularly in later stages of high polyester formation. It is believed that DFK results from a betaketo ester (BKE) byproduct and that the DFK formation rate is only about one thousandth of the sucrose polyester reaction rate.

8. I have examined Exhibit 50, and I confirm that Exhibit 50 comprises accurate copies of pages 107, 108 and 111 of Laboratory Notebook SI 1367 on which I accurately recorded a series of experiments I conducted on February 21-24, and March 2, 1989, respectively. I signed and accurately dated these pages upon the completion of the procedures described on each page respectively. Laboratory Notebook SI 1367 was in my possession and control from June 1988 to July 1989.

9. The experiments described in Exhibit 50 were a part of a matrix of experiments intended to evaluate, inter alia, the effect of potassium hydroxide (KOH) on the sucrose polyester reaction. As described in Exhibit 50 at page 107, lines 5, 15 and 17 and page 108, lines 5, 14 and 20, when the reaction temperature was about 135°F and the potassium soap comprised about 1.1% KOH, sucrose utilization was poor. However, as described at page 111, lines 5, 15 and 19, when the reaction temperature was about 135°F and the potassium soap comprised about 0.14% KOH, sucrose utilization was significantly better. These experiments demonstrated that the

KOH base which accompanies the soap effects the sucrose polyester reaction as set forth in the Appleby et al application at page 14, lines 23-25.

10. I have examined Exhibit 51 attached hereto, and I confirm that Exhibit 51 comprises accurate copies of pages 136-137 and 144-145 of Laboratory Notebook SI 1367 on which I accurately recorded a series of experiments I conducted in May and June 1989, and which I signed and accurately dated upon the completion of the procedures described on each page respectively.

11. Page 136 of Exhibit 51 describes a sucrose polyester reaction which I conducted on May 9, 1989. This reaction was one of several experiments intended to evaluate, inter alia, the effect of temperature on the sucrose polyester reaction. As described in Exhibit 51 at page 136, lines 19, 22 and 31, a later stage of the sucrose polyester reaction was conducted at a temperature of from about 100°C to about 120°C and the resulting sucrose ester product comprised an octaester content of 85% and a DFK level of 87 ppm. The later stage reaction temperature employed in this experiment is within the range disclosed at page 18, lines 6-10 of the Appleby et al Application and the DFK level of the product is within the range set forth at page 24, line 15 of the Appleby et al Application.

12. Page 137 and pages 144-145 of Exhibit 51 describe sucrose polyester reactions which I conducted on May 18, 1989 and June 12-13, 1989, respectively. These reactions were part of a matrix of experiments intended to further evaluate, inter alia, the effect of temperature on the sucrose polyester reaction. As described in Exhibit 51, page 137, line 1, and page 144,

line 2, subsequent reaction stages of the sucrose polyester reaction were performed at a temperature of 100°C. As described at page 145, line 1, the sucrose ester product comprised from about 81% to about 91% octaester and DFK levels of from about 212 ppm to about 374 ppm were obtained. The later stage reaction temperature employed in these reactions is within the range disclosed at page 18, lines 6-10 of the Appleby et al Application and the DFK levels of the products are substantially within the range set forth at page 24, line 15 of the Appleby et al Application.

13. I have reviewed the Declaration Under 37 C.F.R. § 1.608 of Steven R. Alexander and Exhibits Nos. 46-48 and 52 identified in the Alexander Declaration, all of which Declaration and Exhibits are submitted herewith. The Alexander Declaration accurately describes experiments conducted by Mr. Alexander under my direction and control for studying and developing the sucrose polyester process and accurately describes the procedures for recording the general nature and results of such experiments as respectively described therein.

14. As accurately described in the Alexander Declaration, ¶¶ 5-9, and Exhibit 46, a matrix of experiments was performed by Mr. Alexander under my direction and control in September and October of 1989 to investigate the effect of filtration on the sucrose polyester synthesis reaction. I have examined Exhibit 52, and I confirm that Exhibit 52 comprises an accurate copy of page 39 of notebook SI 1386 on which I set forth general instructions for the experiments described in ¶¶ 5-9 of the Alexander Declaration and conducted by Mr. Alexander under my direction and control.

15. In one experiment conducted by Mr. Alexander, the reaction mixture was filtered when the sucrose polyester in the reaction mixture had an I-bar of about 4.6, corresponding to an average degree of esterification of about 57%, to obtain a filtered reaction mixture having an unreacted sucrose (polyol) content of about 0.05% (Alexander Declaration, ¶¶6-7). The average degree of esterification of the sucrose polyester product and the unreacted polyol content of the reaction mixture at the time of the filtration are within the ranges set forth in the Appleby et al Application at page 16, lines 12-17 and lines 35-38, respectively. In another experiment conducted by Mr. Alexander, the reaction mixture was filtered, whereafter both the filtered reaction mixture and an unfiltered control reaction mixture were subjected to further reaction (Alexander Declaration, ¶8). The filtered reaction mixture contained sucrose polyester product comprising about 99% octaester about two hours after the filtration step while the unfiltered reaction mixture contained sucrose polyester product comprising about 77% octaester at about that same time (Alexander Declaration, ¶9), which evidenced the filtered reaction mixture produced octaester significantly faster than the comparable unfiltered reaction, as discussed generally at page 16, lines 12 - 38 of the Appleby et al Application.

16. I have reviewed Exhibit 53 and confirm that it is an accurate copy of the Chemicals Product Development Monthly Report which I prepared on or about November 1, 1989. As described in Exhibit 53, reactions conducted during the previous month further demonstrated that filtration to remove soap, unreacted and burnt sucrose and carbonate solids from the sucrose polyester synthesis reaction mixture enhances the FG (sucrose polyester) reactions as set forth in the Appleby et al application at page 16, line 10-page 17, line 6.

17. I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the present application or any patent issued thereon.

Respectfully submitted,

By: Ju-Nan Kao
Ju-Nan Kao
Date: April 7, 1999

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